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METHODS OF CHEMICAL ANALYSIS OF FERROSILICON

PART III DETERMINATION OF SULPHUR

(*First Revision*)

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METHODS OF CHEMICAL ANALYSIS OF FERROSILICON

PART III DETERMINATION OF SULPHUR

(First Revision)

0. FOREWORD

0.1 This Indian Standard (Part III) (First Revision) was adopted by the Indian Standards Institution on 29 January 1982, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 IS : 1559-1961* which covers chemical analysis of different ferro-alloys, namely, ferrosilicon, ferrochromium, ferromanganese and speigeleisen, silicomanganese, ferromolybdenum, ferrophosphorus, ferrotitanium, ferrovanadium and ferrotungsten, was first published in 1961. While reviewing this standard, the Sectional Committee felt that the standard should be revised to cover ferrosilicon and methods of analysis of other ferro-alloys should be covered in separate standards. During preparation of the revision of IS : 1559-1961*, it was felt convenient to cover methods of chemical analysis of ferrosilicon in different parts of the standard which on publication would supersede Section I ferrosilicon of IS : 1559-1961*. The committee also decided to rewrite the methods taking into consideration the latest composition specified in IS : 1110-1981†. Though calcium and manganese are not specified in IS : 1110-1981†, the determination of these elements are covered in this standard because these are generally required from the viewpoint of determining interference of analysis in methods for determining other elements in ferrosilicon due to presence of these elements. Moreover the determination of these elements may be required for comprehensive analysis of ferrosilicon. This part covers the determination of sulphur. The other parts cover as follows:

Part I	Determination of silicon
Part II	Determination of carbon

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*Methods of chemical analysis of ferro-alloys.

†Specification for ferrosilicon (*third revision*).

Part IV	Determination of phosphorus
Part V	Determination of aluminium
Part VI	Determination of calcium
Part VII	Determination of manganese

0.3 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part III) covers the determination of sulphur (up to 0·050 percent) in ferrosilicon.

2. SAMPLING

2.1 It shall be drawn and prepared in accordance with IS : 1472-1977†.

3. QUALITY OF REAGENTS

3.1 Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1977‡) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4. DETERMINATION OF SULPHUR BY THE GRAVIMETRIC METHOD

4.1 Outline of the Method — The sample is dissolved in nitric and hydrofluoric acids and the solution is finally brought into hydrochloric acid medium. From the solution, sulphur is precipitated as barium sulphate, which is filtered, washed, ignited and weighed.

4.2 Reagents

4.2.1 *Concentrated Nitric Acid* — rd = 1·42 (conforming to IS : 264-1976§).

4.2.2 *Concentrated Hydrochloric Acid* — rd = 1·16 (conforming to IS : 265-1976||).

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*Rules for rounding off numerical values (*revised*).

†Methods of sampling ferro-alloys, for determination of chemical composition (*first revision*).

‡Specification for water for general laboratory use (*second revision*).

§Specification for nitric acid (*second revision*).

||Specification for hydrochloric acid (*second revision*).

4.2.3 *Hydrofluoric Acid* — 40 percent (*v/v*).

4.2.4 *Sodium Carbonate* — Solid (AR).

4.2.5 *Dilute Hydrochloric Acid* — 1:99 (*v/v*).

4.2.6 *Zinc* — 99·9 percent purity.

4.2.7 *Barium Chloride Solution* — 10 percent (*m/v*).

4.2.8 *Acid Wash Solution* — Dilute 5 ml of concentrated hydrochloric acid with water to 1 000 ml and add one gram of barium chloride.

4.3 Procedure

4.3.1 Place 5 g of dried sample into a 600-ml tall from beaker, add 30 ml of concentrated nitric acid and 30 ml of concentrated hydrochloric acid. Add hydrofluoric acid dropwise until the vigorous reaction has subsided. Evaporate to dryness. Add a further 30 ml of concentrated hydrochloric acid, evaporate to dryness, and repeat this operation for two more times.

4.3.2 Finally extract the dried mass in about 10 ml of dilute hydrochloric acid and warm gently until all the salts are dissolved. Add 0·5 g of sodium carbonate, dilute to about 50 ml with hot water and filter through a pulp pad into a 400-ml beaker, washing with dilute hydrochloric acid. The volume of the filtrate at this stage should be about 150 ml.

4.3.3 Add 0·5 g of zinc till the solution is colourless. Filter, wash and add to the filtrate above 20 ml of barium chloride solution. Digest on a steam bath for one hour. Allow to stand overnight. Filter on a pad transferring the entire precipitate from the beaker to the pad with dilute acid wash solution. Wash the pad alternately with the acid wash and distilled water until free from iron salts. Finally wash with distilled water until free from chlorides.

4.3.4 Transfer the pad and precipitate to a platinum or porcelain crucible. Dry and ignite at a low temperature until carbonaceous matter has been removed and finally ignite to constant mass at 900 to 950°C. Cool in a desiccator and weigh.

4.3.5 Carry out a blank determination following the same procedure and using the same quantity of all reagents.

4.4 Calculation

$$\text{Sulphur, percent} = \frac{(A - B) \times 13{\cdot}74}{C}$$

where

A = mass in g of barium sulphate obtained,
B = mass in g of barium sulphate in the blank, and
C = mass in g of the sample taken.